Research Paper | Botany | E-ISSN No : 2454-9916 | Volume : 2 | Issue : 2 | Feb 2016

ANALYSES OF THE VOLATILE OIL CONSTITUENTS OF LANDOLPHIA OWARIENSIS P. BEAUV

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ABSTRACT

The volatile oil, obtained by hydrodistillation of the leaves of *Landolphia owariensis* P. Beauv using Clavenger type apparatus, was analysed for its constituents GC-MS method. The examined material contained 0.08%v/w of essential oil. A total of 34 compounds were identified in the essential oil, accounting for 92.93% of the oil composition. The main components of the essential oil were pentadecanal (13.63%), 1-dodecanol (6.32%), tetradecanol (5.83%), hexadecatrienal (5.62%), squalene (4.63%), ß-ionone (3.25%), α- ionone (2.38%), supraene (3.01%), α-farnesene (3%), carophyllene oxide (2%) and -spathulenol (1.78%). This is the first report on the volatile constituents from the leaves of *Landolphia owariensis*.

INTRODUCTION

The *Landolphia genus* (Apocynaceae), which consist of 63 species, is well distributed from Guinea to West Cameroon and extending across Central Africa to Sudan, Uganda and Southern Tanganyika [1]. This herbaceous plant with a characteristic odour is the only one in West Africa out of the three varieties recognised, and its botanical characters have been described in the literature [2]. In Congo, sap expressed from the leaves is dipped into the eyes and used to wash the patient's face as a treatment for giddiness and epilepsy. In Ghana the acid-pronounced pulp of the fruit is edible and is sometimes used to season food [3]. In some Nigeria localities, decoction of the leaf is used to cure malaria, fever and rheumatic pain (Personal communication, Muazzam Ibrahim 2015). Nwaogu *et al* [4] reported the phytochemical composition of *Landolphia owariensis* leaf extract namely alkaloids, tannins, saponins and flavonoids. *In vitro*, extracts of the root displayed significant antitrypanosomal activity, while only the chloroform extracts of the leaves and stem bark showed activity at 4 and 2 mg/ml]5]. Large amount of fat in the form of oil with agreeable colour and odour which have potentials for use as domestic oil has been reported to be present in the seed extract of *Landolphia owariensis* [6]. It has also been reported that the gas chromatographic analysis on the methyl ester mix of *Landolphia owariensis* string seed pulp gave hexadecanoic acid (palmitic acid) as the principal fatty acid(54.38%). Palmitoleic acid (18.54%) and linoleic acid (9.56%) ranked second and third respectively [7]. However, to our knowledge no phytochemical investigations of the leaves essential oil of *Landolphia owariensis* have been reported to date. Thus this aims to evaluate the chemical constituents of *Landolphia owariensis* leaves essential oil.

MATERIALS AND METHODS

1.1 Chemicals

Hexane and anhydrous sodium sulphate are analytical grade obtained from Sigma-Aldrich (Germany).

1.2 Plant Material

The leaves of *L. owariensis* were collected from Suleja, Niger State, North Central Nigeria in July 2015 by Mallam Muazzam Ibrahim. The identification and authentication of the plant was done by Dr. Grace Ugbabe at the Institute for Pharmaceutical Research and Development, Abuja, Herbarium where the voucher specimen (NIPRD/H/6692) was deposited.

1.3 Essential Oil Isolation

The dried leaves of *L. owariensis* were chopped into small pieces. 400g of the dried plant material was subjected to hydrodistillation using Clevenger type apparatus of 2 litre capacity. One litre of distilled water was added to the material. The mixture was heated on heating mantle at 100 °C. The hydrodistillation was continued for three hours. The light yellow essential oil obtained was dried over anhydrous sodium sulphate and stored at 4 °C in sealed vials until analysis.

1.4 Gas Chromatography-Mass Spectrometry (GC-MS) Analysis

The GC method was then transferred to gas chromatography coupled to mass spectrometry with slight modifications for identification of various phytoconstituents of the essential oil. GC-MS analysis was performed using an Shimadzu GCMS QP-2010 Ultra system (Shimadzu Corporation, Kyoto Japan)) equipped with a fused silica capillary column (60 m x 0.25 mm, i.d.) coated with HP 5 methysilicone (film thickness 0.25 µm) Optima 5ms [Macherey-Nagel GmbH & Co, Germany] and Shimadzu GCMSsolution software. Helium (1.61 ml/min) was used as a carrier gas. The program used for GC oven temperature was isothermal at 60 °C, then it was incremented to 180 °C at a rate of 10°C/min, held at 180 °C for 2 minute, then it was incremented to 280 °C at a rate of 15°C/min, then again held at 280 °C for 4 minutes (Table 1). Injector temperature was 250 °C, ion source temperature was 200 °C and interface temperature was 250 °C. The ionization of sample components was performed in the EI mode (70eV). Individual constituents were identified by referring to compounds known in the literature data [8. Adams RP. Identification of Essential Oil Components by Gas Chromatography / Mass Spectrometry, 4th Ed. Allured Publishing, Carol Stream, Illinois, USA, 2007] and also by comparing their mass spectra with known compounds and NIST Mass Spectral Library(NIST 11), as well as the Flavour and Fragrance Natural and Synthetic Compounds mass spectral library database.

Table 1: The optimized temperature program for column oven

Rise in Temperature per minute (°C)	Temperature (°C)	Hold time (minute)
0	60	0
10	180	2
15	280	4

Injector temperature was 250 °C while ion source temperature was 200 °C and interface temperature was 250 °C. Helium was used as a carrier gas, at a flow rate (1.61 ml/min). Diluted sample (1/100 in hexane, v/v) of 1.0 µl was injected using auto-sampler and in the split mode with split ratio of 50:50.

RESULTS AND DISCUSSION

The light yellow essential oil from *Landolphia owariensis* leaf was obtained in 0.08%v/w yield. The oil was subjected to GC and GC-MS analysis. A total of 50 compounds were detected in the essential oil out of which 34 compounds were identified, accounting for92.93% of the oil composition. The components identified, their retention times and their percentage area are summarized in Table 2, and are arranged in their order of elution on Optima 5ms capillary column. The essential oil from *Landolphia owariensis* leaf showed

diverse composition, it contained the high amount of aliphatic aldehydes. The chemical composition of the essential oil was dominated by hydrocarbons and sesquiterpenes. The most representative class of compounds in the oil was aliphatic hydrocarbons (14%), followed by aromatic compounds (12%), oxygenated sesquiterpenes (10%), aliphatic alcohols (8%), fatty acids (6%), triterpene hydrocarbons (4%), oxygenated monoterpenes (4%), aldehydes (4%), ketones (4%), and small amount of ethers (2%). The major components of the essential oil identified were pentadecanal (13.63%) an aliphatic aldehyde, 1-dodecanol (6.32%), tetradecanol (5.83%), hexadecatrienal (5.62%), squalene (4.63%), 3- buten-2-one (3.25%), α-ionone (2.38%), supraene (3.01%), α-farnesene (3%), carophyllene oxode (2%) and (-)-spathulenol (1.78%). The oil showed presence of important oxygenated monoterpenes such as citronellylacetate and geranylacetone <cis>, as well as the alcohols dodecan-1-ol, tetradecan-1-ol, spathulenol and tridecan-1-ol. Also the oil showed presence of two oxygenated sesquiterpenes namely hexahydrofarnesylacetone and caryophyllene oxide. Pentadecanal has been reported as the major constituent (38.5%) of *Mitracarpus scaber* leaf essential oil [9]. *Coriandrum sativum* L herb had been reported to contain 4.2% of 1- Dodecanol. It is a flavor and fragrance agent [10]. α-*Ionone* is used as a flavour and fragrance agent. It has a floral type odour and a floral type flavour. ß-*Ionone* is a constituent of rose flower (*Rosa damascena*) and is also used in fragrances. It is used in all areas of perfumery. The ionones are derived from the degradation of carotenoids [11].

Table 2: Chemical composition of leaves essential oil from *Landolphia owariensis* P. Beauv

Peak	Compounds	Retention Time	% Composition
1	n.i.	7.617	0.132
2	4α-methyldecalin-1-yl-acetate	8.136	0.40
3	Tridecane	8.285	0.99
4	n.i.	8.445	0.67
5	Citronellyl acetate	8.939	4.05
6	Tetradecane	9.608	2.02
7	α-lonone	9.977	2.38
8	cis-Geranylacetone	10.231	3.30
9	n.i.	10.317	0.12
10	1-Dodecanol	10.521	6.32
11	ß-lonone	10.715	3.25
2	Hexadecane	10.858	1.36
13	α-farnesene	10.956	3.00
14	5,5,8a-Trimethyl-3,5,6,7,8,8a-hexahydro-2H-chromene	11.140	1.19
15	ß-Nerolidol	11.629	1.73
16	n-Tridecan-1-ol	11.733	1.12
17	Supraene	11.813	3.01
18	(-)-spathulenol	11.920	1.78
19	Caryophyllene oxide	12.001	1.99
20	Hexadecane	12.057	2.19
21	n.i.	12.116	0.48
22	n.i.	12.5561	0.16
23	1-Tetradecanol	13.023	5.83
24	n.i.	13.311	0.21
25	Pentadecanal	13.598	13.63
26	6-Phenyldodecane	13.867	1.20
27	Eicosane	14.941	1.34
28	2-Phenyldodecane	15.033	0.92
29	6-phenyl-tridecane	15.309	1.06
30	5-phenyl-tridecane	15.400	1.15
31	Hexahydrofarnesyl acetone	15.496	2.68
32	Benzyl salicylate	15.810	1.88
33	n.i.	15.904	0.63
34	Hexadecatrienal <7,10,13-cis,cis,cis->	16.058	5.62
35	Farnesyl acetone	16.319	2.14
36	Methyl palmitate	16.386	1.68
37	Dibutyl phthalate	16.718	0.92
38	Ethyl palmitate	17.053	1.49
39	Squalene	17.242	4.63
40	Isopropyl palmitate	17.328	1.00
41	n.i.	17.480	0.15
42	n.i.	17.906	0.76
43	n.i.	18.155	0.73
44	n.i.	18.363	0.28
45	n.i.	19.321	0.81
46	n.i.	20.125	0.67
47	n.i.	20.522	0.53
48	Ethylhexyl phthalate	20.835	5.68
49	n.i.	21.705	0.56
50	n.i.	22.030	0.19
	Total	92.93%	
	Oxygenated monoterpenes	4	
	Oxygenated sesquiterpenes	10	
	Triterpene hydrocarbons	4	
	Fatty acids	6	
	Ketones	4	
	Ethers	2	
	Aliphatic hydrocarbons	14	

Key: n.i. = not identified

CONCLUSION

The main components of the essential oil were pentadecanal (13.63%), 1-dodecanol (6.32%), tetradecanol (5.83%), hexadecatrienal (5.62%), squalene (4.63%), 3-buten-2-one (3.25%), α- ionone (2.38%), supraene (3.01%), α-farnesene (3%), carophyllene oxide (2%) and (-)-spathulenol (1.78%). These findings will be helpful in further application of this plant as natural source of pentadecanal and 1-dodecanol.

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